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Versatile sample handling system for scanning tunneling microscopy studies of molecular beam epitaxy

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The ongoing development of short-period semiconductor superlattices for electronic and optoelectronic applications requires atomic-scale control of epitaxial growth, especially at the interfaces.[1] Given this requirement, there is a critical need for *in situ* characterization on the atomic scale as provided by scanning tunneling microscopy (STM).[2] Here we describe a sample handling system designed to integrate a modified commercial STM[3] into a multichamber ultrahigh vacuum (UHV) molecular beam epitaxy (MBE) facility. The system uses a simple, yet versatile, sample holder design that enables quick and easy sample transfers between multiple chambers, including two Riber MBE and two surface characterization chambers interconnected by Riber UHV ModuTrac™.

The chief component of the sample handling system is a molybdenum sample holder, shown in Fig. 1(a). The essential elements of the holder are its square shape and two pairs of clearance holes oriented at right angles to each other: one pair through the front near the top of the holder, and the other running from top to bottom along the edges. As will be described below, the holes are for mounting the holder on the UHV manipulators and STM sample stage. As long as these four holes are present, the basic holder can be modified in a number of ways depending on the sample mounting and heating requirements. The version shown in Fig. 1 is a ''window frame'' design with four tapped holes through the face of the holder that are used to mount a semiconductor wafer sample. The interior cutout permits direct radiative heating of the wafer sample along with infrared transmission-based thermometry during MBE.[4] Alternately, the holder can be left solid if heating of the wafer via thermal contact with the holder is desired. Note that mounting the wafer sample on the front face of the holder allows grazing-angle access for reflection high energy electron diffraction (RHEED).

The sample holder mounts onto a commercial STM (with a modified sample stage) by inserting the two vertical pins on the STM stage into the long holes running the length of the holder [Fig. 1(b)]. The pins on the STM are of unequal length, with the longer pin having raised points near the top and bottom, and the shorter pin having a raised point near the top end only [see Fig. 2(c)]. Two identical molybdenum spring clips (0.25 mm thick) push on the front of the sample holder, holding the inside of the long holes against the three raised points on the pins and thereby achieving a stable contact. In addition to the standard 1 cm of horizontal sample motion available, this modified design permits the sample to be stably mounted up to 5 mm above the base, resulting in a viewable area of ~ 0.5 cm^2. To perform MBE on an STM-compatible sample, the sample holder is mounted on a standard 5-cm-diam ''moly-block'' [Fig. 1(c)]. The sample holder is held in place by two 0.25-mm-thick molybdenum compression clips screwed to the moly-block (the only required modifications of the moly-block). Screws made of tantalum are used to avoid seizing in the moly-block.

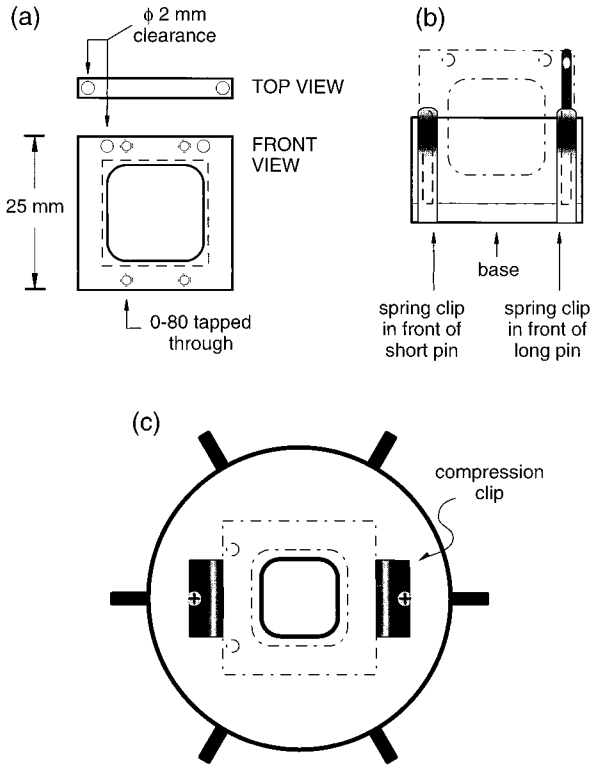

FIG. 1. (a) Schematic drawing of a ''window frame'' design sample holder. Samples up to 17 mm×17 mm (dashed square) can be mounted onto the front by four 0–80 screws. (b) Front view of the modified sample stage on the STM. The sample holder mounts onto two pins, one shorter than the other, and is held in place by molybdenum spring clips. The raised area near the top of the long pin is shown as a white spot. Outlines of the sections of the pins hidden behind the clips are shown as dashed lines. The dash–dot–dash lines show the outline of a sample holder when mounted. See Fig. 2(c) for a side view. (c) Moly-block used for MBE. The sample holder is held onto the face by molybdenum compression clips attached to the moly-bock by tantalum screws. All components are drawn approximately to scale.

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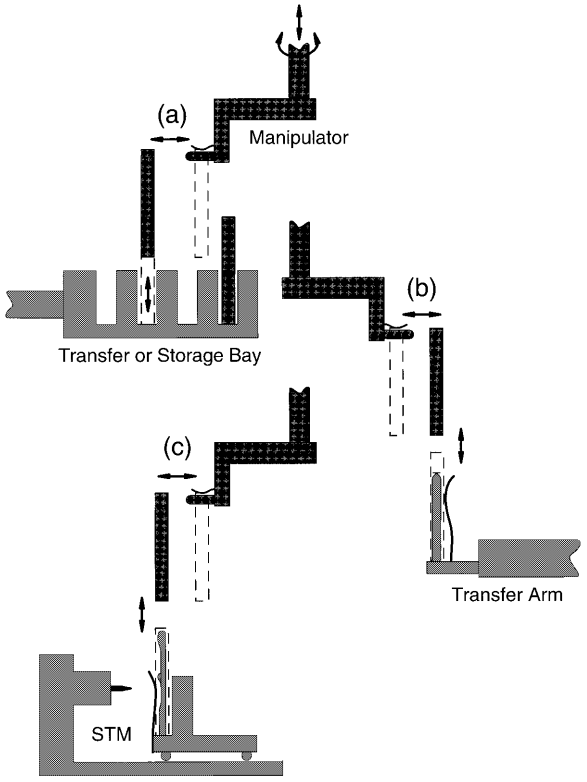

FIG. 2. Illustrations (side view) of the sample transfers between the manipulator and (a) the load-lock transfer bay or sample storage bay; (b) the ModuTrac transfer arm; (c) the STM. The dashed lines show the outline of the sample holder in the mounted positions.

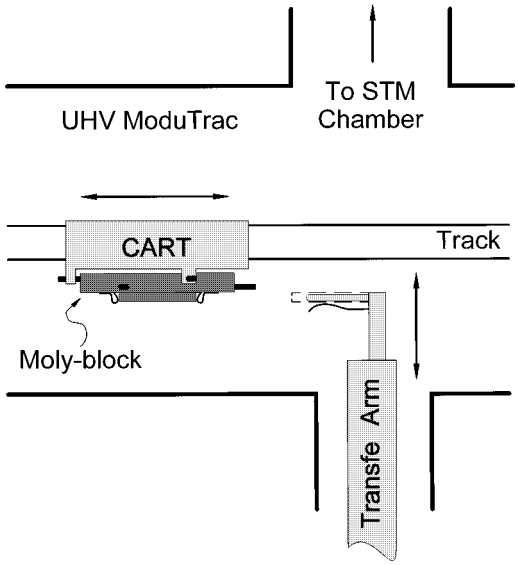

FIG. 3. Top view (inside the ModuTrac) of the motions for transfer of a sample holder between the ModuTrac transfer arm and a moly-block mounted on a cart in the ModuTrac. Note that the track is on the bottom of the ModuTrac chamber.

In addition to the STM and the moly-block, the sample holder also mounts on an UHV sample manipulator, a load-lock transfer bay, a sample storage bay, and a ModuTrac transfer arm (all except the last are on the STM chamber), as shown in Fig. 2. The sample holder mounts onto the sample manipulator via two horizontal pins that are inserted into the holder from the back, and is locked into place by a spring clip that pushes down on the top edge. The load-lock transfer bay and sample storage bay both have a slotted-plate design, holding four and seven sample holders, respectively, and are attached to magnetically coupled linear-rotary transporters. (Sample holders rest upright in slots, held in place by gravity.) The ModuTrac transfer arm, also a magnetically coupled linear-rotary transporter, has a simple pair of pins and spring clips on the end, oriented perpendicular to the axis of rotation. These pins and clips engage the long holes in the sample holder in a manner similar to those on the STM stage.

Transfer of the sample between the various components within the multichamber facility relies on perpendicular motions between the sample manipulators and the sample holder mounts, as indicated in Fig. 2. Transfer from the manipulator to the load-lock transfer bay or sample storage bay occurs by first positioning the empty bay slot under the sample holder, then lowering the manipulator to place the sample holder into the slot, and finally translating the bay

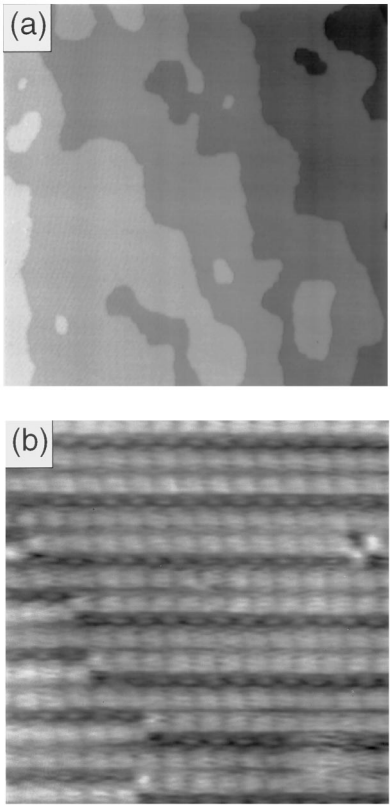

FIG. 4. Filled-state STM images of a 1-μm-thick GaSb film grown by MBE on a GaSb(001) substrate. (a) 160 nm$\times$160 nm area, with each gray level representing a change in height of 0.3 nm (a single GaSb layer). (b) 13 nm$\times$13 nm area showing the atomic-scale $2\times5/c(2\times10)$ reconstruction.

laterally in order to disengage the sample holder from the manipulator. To transfer the holder onto the manipulator from one of the bays, the reverse procedure is used. Transfers between the manipulator and ModuTrac transfer arm are performed in a similar fashion. Transfers between the manipulator and STM occur via motion of the manipulator only, utilizing the 25 mm of in-plane motion permitted by the manipulator micrometers.

The essential components required to transfer the sample holder from the STM chamber to the moly-block (and subsequently to a MBE chamber) are illustrated in Fig. 3. Following transfer from the manipulator to the ModuTrac transfer arm, the sample is rotated 90° and then pulled out of the STM chamber to the far side of the ModuTrac. The moly-block cart, which runs on a track down the center of the ModuTrac, is then positioned in front of the transfer arm. The sample holder (on the end of the transfer arm) is then pushed into the moly-block, engaging the holder between the compression clips. Finally, the cart is moved off to the left, disengaging the sample holder from the transfer arm. Transfer from the moly-block to the STM chamber occurs with the reverse procedure. Note that the simple two-clip mounting system on the moly-block provides considerable latitude in the alignment of the sample holder during these transfers.

The effectiveness of the sample holder design for *in situ* study of MBE-grown films is demonstrated by the images shown in Fig. 4 of a GaSb(001) homoepitaxial surface.[5] Both the large-scale surface morphology and the atomic-scale structure can be readily observed after cooling the sample to ambient temperature.

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